INDUSTRIAL HYGIENE EQUIPMENT CALIBRATION

THE H-NU PHOTOIONIZER

MIRAN 1A INFRARED PHOTOSPECTROMETER

J.KENNY, 1-92

INTRODUCTION:

The H-NU photoionizer and MIRAN 1A infrared photospectrometer finds many uses at Fermilab in measuring airborne hazards associated with organic solvents. Annual calibrations of these devices are necessary to ensure accurate field measurements.

Successful calibration of the H-NU requires a good working knowledge of the instrument through its use in the field. It is recommended that the person performing the calibration read all the H-NU literature and perform a practice calibration to familiarize him/herself with the process.

READ THIS GUIDE BEFORE STARTING A CALIBRATION.

EQUIPMENT:

1. H-NU photoionizer, with fully charged battery and both (10.2 & 11.7eV) probes and/or Foxboro Miran 1A infrared spectrometer.

2. Approximately 5 feet of 3/8-inch tygon or rubber tubing to connect the MIRAN inlet and outlet with the chamber.

3. Calibration chamber, including exhaust and mixing fans, hot plate, and ring stand

in the labeled box. DO NOT use any other equipment without the permission of the owner.)

7. A thermometer for temperature readings inside the chamber.

8. Tape and parafilm to seal the calibration chamber and hose connections.

9. A rubber bulb for pipetting.

10. A calculator.

11. Duct tape.

12. H-NU/MIRAN literature and documentation.

PROCEDURES:

H-NU/MIRAN calibrations take time. Since the H-NU and MIRAN calibration procedures are similar, the two devices should be calibrated simultaneously. Those performing calibrations should be somewhat familiar with basic chemical laboratory techniques.

Use a copy of the *H-NU/MIRAN Calibration Checklist* found in Appendix C for each calibration. Place completed checklists in the respective device file binders.

1. Before all calibrations, note and record the air temperature in the chamber.

2. •(H-NU) Install the H-NU with the probe inside the chamber. Firmly fasten the probe to the ring stand near the chamber center. Seal the probe access hole with tape.

•(MIRAN) Attach inlet and outlet hoses to the MIRAN and thread both into the chamber access hole. Inside the chamber, place an organic vapor cartridge on the ring stand mount and attach it to the end of the inlet hose. Seal the MIRAN hose connections with parafilm and the chamber access hole with tape.

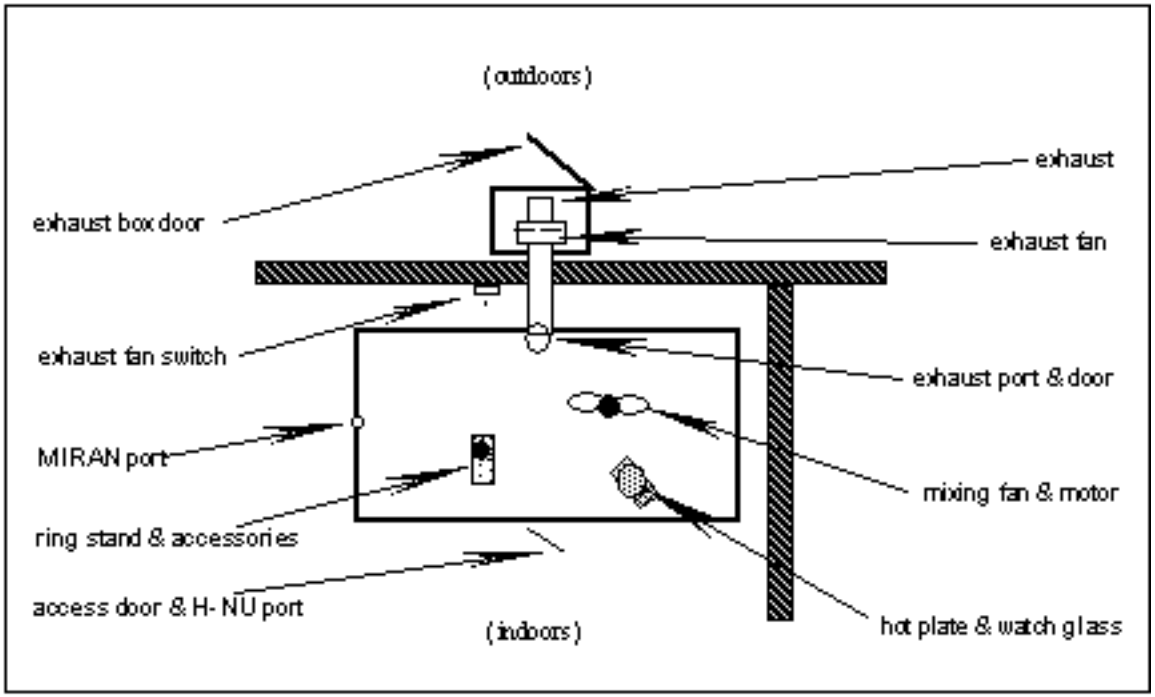

Figure 1 - Top cut-away view of 21 Shabbona calibration chamber

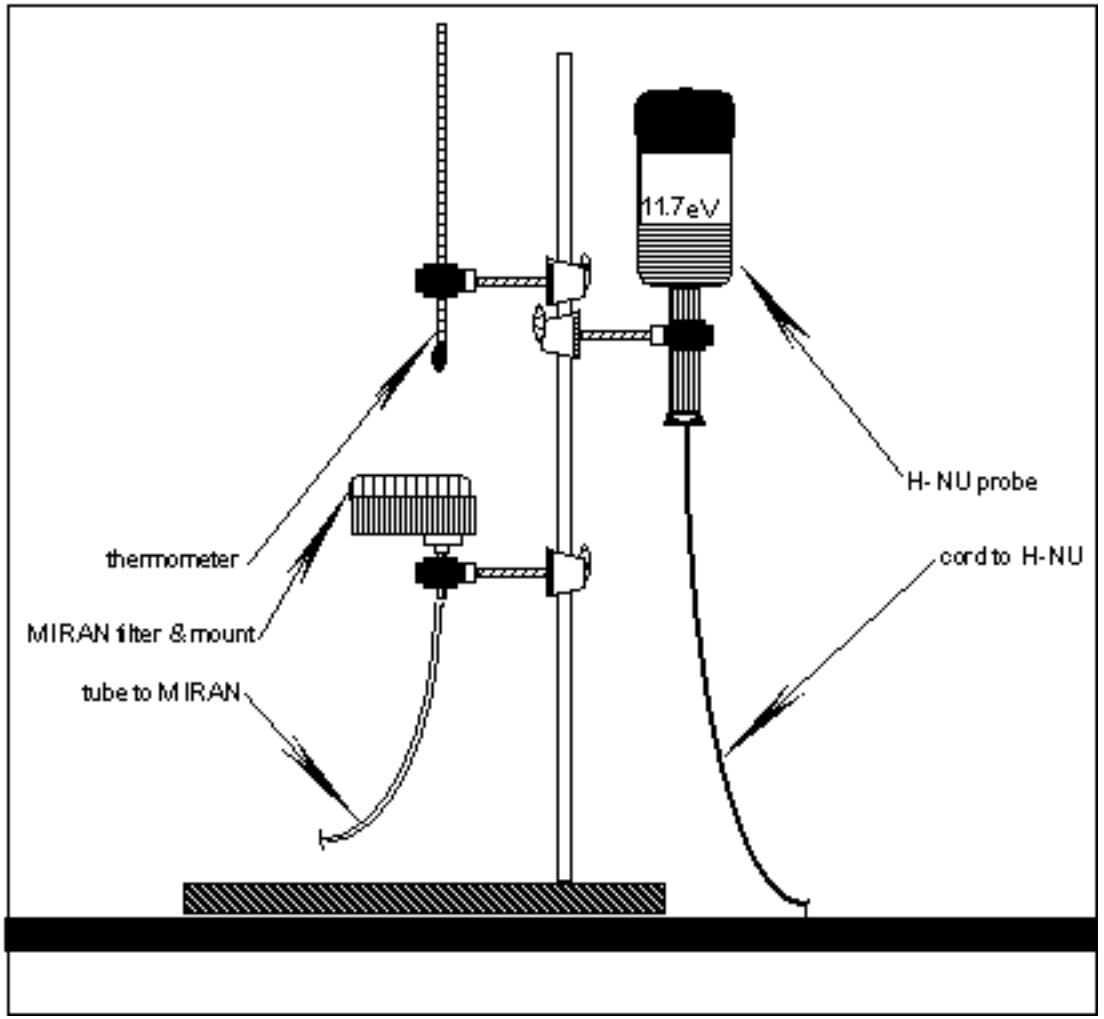

Figure 2 - Acceptable setup of ringstand with MIRAN and H-NU hardware

NOTE: During simultaneous H-NU/MIRAN calibrations, the H-NU should be placed on a chair near the front chamber door. The small hole near the edge of this door should then be used to allow H-NU probe access to the chamber.

3. •(H-NU) Turn on the H-NU to **STANDBY** and allow five minutes for warm-up.

•(MIRAN) Open the MIRAN inlet and outlet vents and turn on both the MIRAN main power and fan. Allow a five minute warm-up.

6. Open several 21 Shabbona doors and windows to help clear interfering factors from the lab air.

7. While the H-NU and/or MIRAN warm up, calculate volumes needed for solvent additions to the chamber. (See Appendix A.)

8. •(H-NU) Turn the **ZERO** knob with the H-NU on **STANDBY** until the response meter reads **0.** Turn the H-NU to **0-20** and record the meter response as <u>background.</u> This background reading should be subtracted from all subsequent readings.

•(MIRAN) Set the MIRAN to the recommended path- and wavelength for the chosen calibration factor. Then turn the **RANGE** knob to **0.025** and use the **zero** knob to zero the device. It may be necessary to flip the **1x/10x** switch found just to the right of the **zero** knob to bring the needle into the proper range for zeroing.

9. Turn off the exhaust fan and close the exhaust port door.

•(MIRAN) Replace the organic filter cartridge on the inlet hose with a HEPA filter. Store the organic vapor cartridge in a sealed ziploc-type bag.

10. Close the chamber front door.

11. Remove a bottle of the solvent from the yellow cabinet and pour a very small amount into a 30mL beaker.

12. Immediately cover the beaker with a watch glass to reduce evaporation.

13. •(H-NU) SKIP THIS STEP UNLESS STARTING A CALIBRATION FOR A FACTOR ON THE H-NU. The first calibration point for a given factor on the H-NU should be done at the ACGIH threshold limit value (TLV) to find the proper span setting. Add to the chamber the amount of solvent (see Appendix A) that will evaporate into the TLV for that factor and turn on the hot plate and mixing fan until a stable reading is noted. Turn off the fan and hot plate. Turn the **SPAN** knob until the TLV or a factor of ten of the TLV is noted on the meter. (This may require adjustment of the **RANGE** selector.) Record the span setting and x-factor. (The x-factor is the number by which the meter-face reading must be multiplied to get the actual concentration.) Clear the chamber for 10 minutes, reseal the chamber, and proceed using the new span setting and x-factor.

14. Add the amount of solvent (to the watch glass on the chamber hot plate) that will evaporate to one-half the TLV. Initialize mixing fan and watch for the solvent to evaporate. Some solvents may require heating to evaporate entirely. (If necessary

18. Add the amount of solvent that will raise the solvent's chamber concentration to the TLV.

Start the mixing fan and hot plate if necessary.

19. Allow the chamber air to mix for five minutes.

20. Stop the mixing fan

21. Take and record the second reading.

22. Add the amount of solvent that will raise the solvent's chamber concentration to twice the TLV.

23. Start the mixing fan and hot plate if necessary.

24. Allow the chamber air to mix for five minutes.

25. Stop the mixing fan

26. Take and record the third and final reading.

27. Clear the chamber for 10 minutes before starting the next calibration. Spend this time making a simple graph of your data, with actual chamber solvent concentration versus concentrations shown by the H-NU and or MIRAN. Redo calibrations which yield grossly nonlinear plots. Plots for most solvents should be linear to ~300 ppm on both devices.

28. When finished calibrating for a day, return the lab to the state in which you found it. This takes approximately 45 minutes, so plan ahead.

HINTS

1. Rotate pipettes. Keep one set in a warm place to evaporate residual leftover solvent while using the other set.

2. Handle syringes briefly and only with their flanges and plungers. A micro syringe at body temperature holds less than one at room temperature.

3. Remove the plungers from the syringes and allow both barrels and plungers to dry for one hour before storing for long periods.

Appendix A

Densities and molecular weights of solvents used in H-NU/MIRAN calibrations.

Factor	Density	Molecular Weight	µL/ppm*
acetone	0.79g/cc	58	2.98
chloroform	1.50	120	3.26
methyl ethyl ketone	0.81	72	3.62
dimethyl formamide	0.94	73	3.13
ethanol	0.79	46	2.35
	formaldehyde(as formalin) - Varies with concentration; perform calculations with data found on reagent bottle. Use only fresh, reagent grade formalin for calibrations.		
Freon 113	1.58	187	4.81
isopropanol	0.78	60	3.12
methanol	0.80	32	1.62
Stoddard/naphtha	0.97	99	4.13
xylenes	0.87	106	4.93

Appendix B

Calculating needed solvent volumes for H-NU/MIRAN calibrations.

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Using the formula:

$$C(\text{actual}) = \frac{(\text{XmL solv.})(\mathbf{r})(22.4\ \text{L/mol})(1000\text{mg/g})}{(\mathbf{MW})(990\ \text{L})(\mathbf{P}/760\text{mmHg})(273\text{K}/\mathbf{T})}$$

Where:

	r	=	solvent density
	MW	=	solvent molecular weight
	P	=	atmospheric pressure at time of calibration (mmHg)
	T	=	air temperature in lab at time of calibration (K)
	C	=	chamber concentration

ACGIH TLV.

E.g., for 1,1,1-trichloroethane (TLV=350ppm) we choose the concentrations 1, 5, 10, 50, 100, 350, 500, and 1000 ppm. Using the formula above, we calculate the volumes needed to provide these airborne concentrations when the temperature is 24ºC (75ºF) and air pressure is

29.72"Hg (755 mmHg):

$$X\ \mu L/ppm = \frac{(0.0151\ mol\ K/mL\ mmHg)(133.5\ g/mol)(755\ mmHg)}{(1.34 g/mL)(297K)}$$

$X = 3.82\ \mu L/ppm$

So, to make 1 ppm in the chamber, 3.82 mL must be added. For 5 ppm, we need a total of 3.82 x 5 = 19.1 mL. But since the chamber already contains 3.82 mL, we need only add 15.3 mL. A 10 ppm concentration requires the addition of another 19.1 mL 1,1,1-trichloroethane, etc.

Appendix C(MIRAN)

MIRAN CALIBRATION CHECKLIST: Place completed checklist in the MIRAN file binder.

Calibration for:_____

Date: _____

Time: _____

Name & ID#

Chamber Temp._____K Atmospheric Pressure_____mmHg

3. ___ Attach inlet and outlet hoses to the MIRAN and thread both into the chamber access hole.

4. ___ Inside the chamber, place an organic vapor cartridge on the ring stand mount and attach it to the end of the inlet hose.

5. ___ Seal the MIRAN hose connections with parafilm and the chamber access hole with tape.

6. ___ Open the MIRAN inlet and outlet vents and turn on both the MIRAN main power and fan. Allow a five minute warm-up.

7. ___ To clear calibration chamber, open fan box(outdoors), exhaust port, and chamber front door; and run exhaust fan for ten minutes.

8. ___ Open doors and windows to help clear interfering factors from the lab air.

9. ___ Calculate volumes needed for solvent additions to the chamber. (See Appendix A of the H-NU/MIRAN calibration procedures)

Volume for 1/2 TLV: _____µl

Volume for TLV: _____µl

Volume for twice TLV: _____µl

10. ___ Set the MIRAN to the recommended path- and wavelength for the chosen calibration factor.

11. ___ Turn the **RANGE** knob to **0.025** and use the **zero** knob to zero the device.

15. ___ Remove a solvent from the yellow cabinet. Pour a small amount into a 30mL beaker, and cover beaker with a watch glass to reduce evaporation.

16. ___ Add the amount of solvent (to the watch glass on the chamber hot plate) that will evaporate to one-half the TLV of the solvent in use.

17. ___ Initialize mixing fan and watch for the solvent to evaporate.

NOTE: Some solvents may require heating to evaporate entirely. (If necessary, provide for changes in chamber air temperature caused by the hot plate on the checklist.) Turn off the hot plate as the last bit of solvent evaporates.

New Chamber Temperature (if different from (1.), above): _____K

18. ___ Stop the chamber mixing fan after approximately five minutes.

19. ___ Record the meter reading.

NOTE: Record readings only when the needle remains relatively motionless for >15 seconds.

FIRST READING: _____

SECOND READING: _____

THIRD READING: _____

20. ___ Add the amount of solvent that will raise the solvent's chamber concentration to the TLV.

21. ___ Repeat steps 14-16.

22. ___ Add the amount of solvent that will raise the solvent's chamber concentration to twice the TLV.

found it. Be sure to turn off hot plate and return equipment to proper areas.

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Appendix C(H-NU)

H-NU CALIBRATION CHECKLIST: Place completed checklist in the H-NU file binder.

Calibration for:_____

Date: _____

Time: _____

Name & ID#: _____

1. ___ Be sure all equipment needed for calibration is present and operable.

2. ___ Note and record the air temperature in the calibration chamber. Also record the barometric pressure in the area.

Chamber Temp._____K Atmospheric Pressure_____mmHg

3. ___ Fasten the H-NU probe to the ring stand near the chamber center.

NOTE: During simultaneous H-NU/MIRAN calibrations, the H-NU should be placed on a chair near the front chamber door. The small hole near the edge of this door should then be used to allow H-NU probe access to the chamber.

4. ___ Turn on the H-NU to **STANDBY**, and allow five minutes for warm-up.

5. ___ To clear calibration chamber, open fan box(outdoors), exhaust port, and chamber front door; and run exhaust fan for ten minutes.

Volume for 1/2 TLV: _____ µl

Volume for TLV: _____ µl

Volume for twice TLV: _____ µl

8. ___ With H-NU on **STANDBY**, turn the **ZERO** knob until the response meter reads **0.**

9. ___ Turn H-NU to **0-20,** and record the meter response as background.

background _____

NOTE: This background reading should be subtracted from all subsequent readings.

10. ___ Turn off the exhaust fan, and close the exhaust port door.

11. ___ Close the chamber front door, and seal the probe access hole with tape.

12. ___ Remove a solvent from the yellow cabinet. Pour a small amount into a 30mL beaker and cover beaker with a watch glass to reduce evaporation.

13. ___ The first calibration point for a given factor on the H-NU should be done at the ACGIH threshold limit value (TLV) to find the proper span setting. Add to the chamber the amount of solvent (see Appendix A) that will evaporate into the TLV for that factor and turn on the hot plate and mixing fan until a stable reading is noted. Turn off the fan and hot plate. Turn the **SPAN** knob until the TLV or a factor of ten of the TLV is noted on the meter. (This may require adjustment of the **RANGE** selector.) Record the span setting and x-factor. (The x-factor is the number by which the meter-face reading must be multiplied to get the actual concentration.) Clear the chamber for 10 minutes, reseal the chamber, and proceed using the new span setting and x-factor.

New Span Setting: _____

NOTE: Some solvents may require heating to evaporate entirely. (If necessary, provide for changes in chamber air temperature caused by the hot plate on the checklist.) Turn off the hot plate as the last bit of solvent evaporates.

New Chamber Temperature (if different from (1.), above): _____K

16. ___ Stop the chamber mixing fan after approximately five minutes.

17. ___ Record the meter reading.

FIRST READING: _____

SECOND READING: _____

THIRD READING: _____

NOTE: Record readings only when the needle remains relatively motionless for >15 seconds.

18. ___ Add the amount of solvent that will raise the solvent's chamber concentration to the TLV.

19. ___ Repeat steps 14-16.

20 ___ Add the amount of solvent that will raise the solvent's chamber concentration to twice the TLV.

21. ___ Repeat steps 14-16.

22. ___ Clear chamber for 10 minutes before starting the next calibration.

NOTE: Spend this time making a simple graph of your data, with actual chamber solvent concentration versus concentrations shown by the H-NU. Redo calibrations which yield grossly nonlinear plots. Plots for most solvents should be linear to ~300 ppm.

23. ___ When finished calibrating for a day, return the lab to the state in which you found it. Be sure to turn off hot plate and return equipment to proper areas.